15

20

25

CLAIMS

- Process for mixing a first acidic aqueous solution comprising hydroxylammonium and phosphate with a second acidic aqueous solution comprising nitric acid, resulting in a third acidic aqueous solution comprising hydroxylammonium, phosphate and nitric acid, wherein in the third acidic aqueous solution the total acid concentration minus the phosphate concentration is lower than 0.523 * ln([hydroxylammonium]/1.25) + 422/(T + 81) whereby [hydroxylammonium] is the concentration of hydroxylammonium in the third acidic aqueous solution, T is the temperature of the third acidic aqueous solution expressed in °C and all concentrations are expressed in mol/l.
 - 2. Process according to claim 1, wherein $(c_{acid}(1)^*V_1 + c_{acid}(2)^*V_2)/(V_1+V_2) (c_{phosphate}(1)^*V_1 + c_{phosphate}(2)^*V_2)/(V_1+V_2) \\ < 0.523 * ln(((c_{hyam}(1)^*V_1 + c_{hyam}(2)^*V_2)/(V_1+V_2))/1.25) + 422/(T(3) + 81) \\ \text{wherein}$

 $c_{acid}(1)$ and $c_{acid}(2)$ are the total acid concentration in the first acidic aqueous solution and in the second acidic aqueous solution respectively, expressed in mol/l. $c_{phosphate}(1)$ and $c_{phosphate}(2)$ are the phosphate concentration in the first acidic aqueous solution and in the second acidic aqueous solution respectively, expressed in mol/l.

 $c_{hyam}(1)$ and $c_{haym}(2)$ are concentration hydroxylammonium in the first acidic aqueous solution and in the second acidic aqueous solution respectively, expressed in mol/l. T(3) is the temperature of the third acidic aqueous solution.

 V_1 and V_2 are the volume of the first acidic aqueous solution and second acidic aqueous solution respectively.

- 3. Process according to claim 1 or claim 2, wherein the process comprises adding hydroxylammonium to an acidic aqueous solution comprising hydroxylammonium to obtain the first acidic aqueous solution.
- Process according to claim 3, wherein the acidic aqueous solution to which
 hydroxylammonium is added is an aqueous reaction medium leaving a cyclohexanone oxime synthesis reactor in which cyclohexanone oxime is produced by reaction of hydroxylammonium with cyclohexanone.
 - 5. Process according to claim 4, wherein the aqueous reaction medium leaving the cyclohexanone oxime synthesis reactor is separated into at least a first part and a

20

25

second part; and wherein the process comprises adding said hydroxylammonium to the first part of the aqueous reaction medium to obtain the first acidic aqueous solution; and absorbing and/or oxidizing nitrogen oxides in the second part of the aqueous reaction medium to prepare nitric acid.

- 5 6. Process according to any one of claims 1 to 5, wherein the process comprises adding hydroxylammonium to the third acidic aqueous solution.
 - 7. Process according to any one of claims 3 to 6, wherein an aqueous reaction medium leaving a hydroxylammonium synthesis reactor is used to add said hydroxylammonium to said acidic aqueous solution.
- 10 8. Process according to any one of claims 1 to 7, wherein the process comprises feeding the third acidic aqueous solution to a hydroxylammonium synthesis reactor in which hydroxylammonium is prepared by catalytic reduction of nitrate with hydrogen.
- 9. Process according to any one of claims 1 to 8, wherein the third acidic aqueous solution is mixed with an acidic aqueous solution comprising nitric acid, said mixing preferably being carried out at a temperature between 20 and 80 °C, resulting in a fourth acidic aqueous solution comprising hydroxylammonium, phosphate and nitric acid wherein

 $c_{acid}(4)-c_{phosphate}(4) < 0.523*In(c_{hyam}(4)/1.25) + 422/(T(4) + 81)$ wherein $c_{acid}(4) = total$ acid concentration in the fourth acidic aqueous solution, expressed in mol/l

 $c_{phosphate}(4)$ = phosphate concentration in the fourth acidic aqueous solution, expressed in mol/I

c_{hyam}(4) = concentration hydroxylammonium in the fourth acidic aqueous solution, expressed in mol/l

- T(4) = temperature in the fourth acidic aqueous solution expressed in °C.
- 10. Process according to claim 9, wherein the process comprises feeding the fourth acidic aqueous solution to a hydroxylammonium synthesis reactor in which hydroxylammonium is prepared by catalytic reduction of nitrate with hydrogen.
- Process according to claim 9 or claim 10, wherein the process comprises adding hydroxylammonium to the third acidic aqueous solution.
 - 12. Process according to any one of claims 1 to 11, wherein an aqueous reaction medium leaving a hydroxylammonium synthesis reactor is used to add said hydroxylammonium to the third acidic aqueous solution.

WO 2004/052780 PCT/NL2003/000877

- 23 -

- 13. Process according to claim 1 or claim 2, wherein the mixing of the first acidic aqueous solution and second acidic aqueous solution is performed in a hydroxylammonium synthesis reactor in which hydroxylammonium is prepared by catalytic reduction of nitrate with hydrogen.
- Process according to claim 9, wherein the mixing of the third acidic aqueous solution and said acidic aqueous solution comprising nitric acid is performed in a hydroxylammonium synthesis reactor in which hydroxylammonium is prepared by catalytic reduction of nitrate with hydrogen.
- 15. Process according to any one of claims 1 to 14, wherein the process comprises cycling an aqueous reaction medium from a hydroxylammonium synthesis reactor in which hydroxylammonium is prepared by catalytic reduction of nitrate with hydrogen to a cyclohexanone oxime synthesis reactor in which cyclohexanone oxime is produced by reaction of hydroxylammonium with cyclohexanone and from the cyclohexanone oxime synthesis reactor back to the hydroxylammonium synthesis reactor.
 - 16. Process according to any one of claims 1 to 15, wherein an aqueous reaction medium leaving a cyclohexanone oxime synthesis reactor is used as first acidic aqueous solution.
- 17. Process according to any one of claims 1 to 16, wherein the second acidic aqueous solution is obtained by absorbing and oxidizing nitrogen oxides in an aqueous solution.
 - 18. Process according to any one of claims 1 to 17, wherein said mixing of the first acidic aqueous solution with the second acidic aqueous solution is carried out at a temperature between 20 and 80 °C.

25